

### **Outline**

- Application and History
- Chemistries
- Powder Analysis
- Consolidation
- Thermophysical Testing
- Future Testing

### **Rocket Engine Thrust Cell Liner**



MSFC Produced VPS Cu-8 Cr-4 Nb Liner Prior To Close-out

- Thrust cell liners contain the combustion of hydrogen and oxygen in the engine
- Flame temperature is approximately 2760°C (5000°F)
- Liners are actively cooled with cryogenic hydrogen
- Thickness of cooling channel wall is typically near 1 mm (0.040")
- Material subjected to extreme thermal gradients and heat fluxes

## **History**

- Original Development at NASA Glenn Research Center (GRC)
  - Powder lots produced by Special Metals in 1990 and 1992
  - Material properties were greatly superior to NARloy-Z (Cu-3 Ag-0.5 Zr)
    - Exception was somewhat lower thermal conductivity
- Reusable Launch Vehicle (RLV) NRA 8-21 Awarded June 1998
  - Develop database for Cu-8 Cr-4 Nb powder produced by Crucible Research
- Crucible Research Production Runs
  - Three sets of runs have been made
    - First two for Rocketdyne Division of Boeing
    - Third set of runs for NASA GRC and Marshall Space Flight Center (MSFC)
      - Seven runs for GRC each producing ~125 pounds of powder
      - Four runs for MSFC each producing ~125 pounds of powder

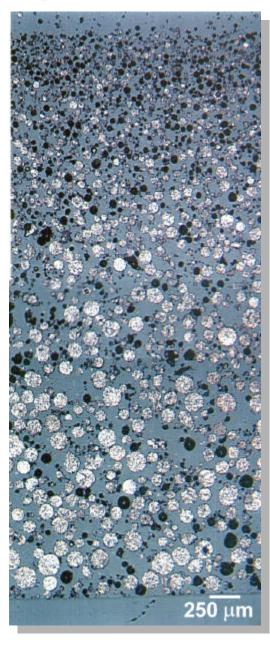
### **Cu-8 Cr-4 Nb Powder Chemistries**

	Cr		Nb		Cr:Nb Ratio		Cr+Nb		Calculated	0
Powder Lot	wt.%	at.%	wt.%	at.%	wt.%	at.%	wt.%	at.%	Cr₂Nb vol.%	ppm
3A	6.80	8.34	5.93	4.07	1.15	2.05	12.73	12.41	14.55	732
3B	6.52	8.00	5.81	3.99	1.12	2.01	12.33	11.99	14.10	711
3C	6.72	8.24	5.89	4.04	1.14	2.04	12.61	12.29	14.42	471
3D	6.78	8.32	5.98	4.11	1.13	2.03	12.76	12.42	14.59	626
3E	6.79	8.33	5.99	4.11	1.13	2.03	12.78	12.44	14.23	476
Special Metals	6.45	7.91	5.79	3.98	1.11	1.99	12.24	11.89	14.00	455
Average	6.72	8.25	5.92	4.06	1.13	2.03	12.64	12.31	14.38	603.2
Std. Dev.	0.12	0.14	0.07	0.05	0.01	0.02	0.19	0.19	0.21	124.9
Minimum	6.52	8.00	5.81	3.99	1.12	2.01	12.33	11.99	14.10	471.0
Maximum	6.80	8.34	5.99	4.11	1.15	2.05	12.78	12.44	14.59	732.0

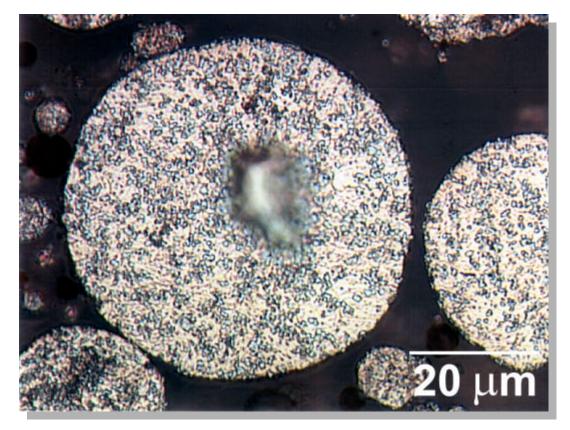
Statistics apply only to the five Crucible Research Cu-8 Cr-4 Nb powder lots

Calculated volume percent Cr<sub>2</sub>Nb uses FCC phase for Cr<sub>2</sub>Nb and assumes all Cr and Nb used

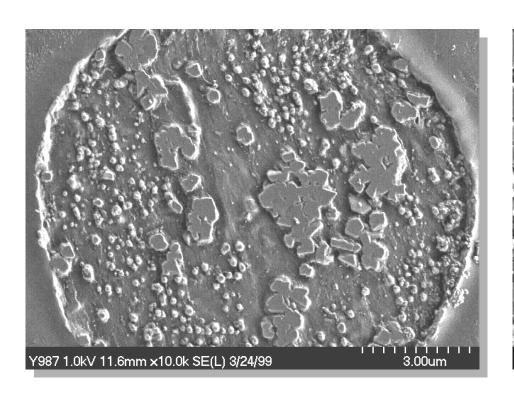
## **Optical Micrograph of Powder Particles**

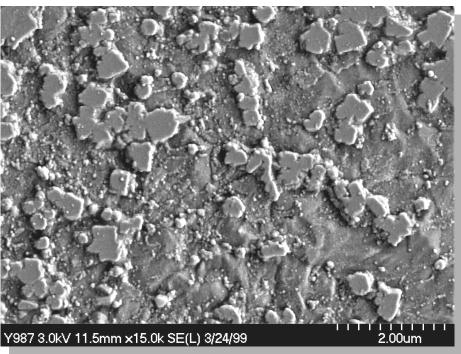


- Powder particles are spherical with few satellites
- Voids are present within many particles
  - Believed to be solidification shrinkage rather than trapped Ar



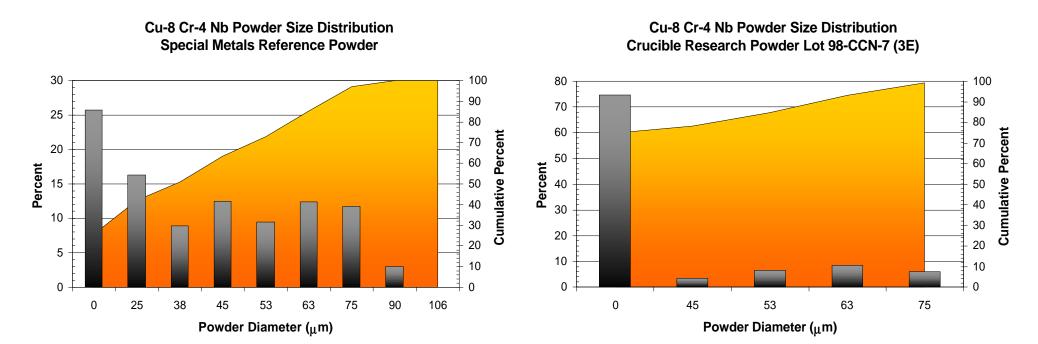
## Cr<sub>2</sub>Nb Precipitates In Cu-8 Cr-4 Nb Powder





- Cr<sub>2</sub>Nb particles appear to be agglomeration of individual precipitates
  - Cr<sub>2</sub>Nb forms in liquid prior to Cu solidification and agglomerates
  - Consistent with Special Metals powders

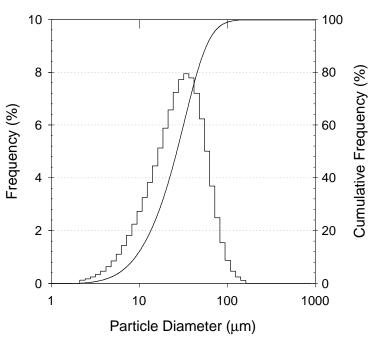
# Powder Size Analysis - Sieve Analysis



- Powder sieved to -150 mesh (<106 μm) to remove any coarse particles
- Over 70% of Crucible Research powder was -325 mesh (<44.5  $\mu$ m) vs. 50% for Special Metals powder

# Powder Size Analysis -Horiba Particle Size Analyzer

Powder Size Distribution Cu-8 Cr-4 Nb Lot 3B (2 min Ultrasonic Agitation)



Powder Lot	Mean Particle Size (μm)
3B	30.265 (σ=22.393)
3C	41.949 (σ = 30.638)
3D	42.784 (σ = 33.816)
3E	36.294 (σ = 29.650)

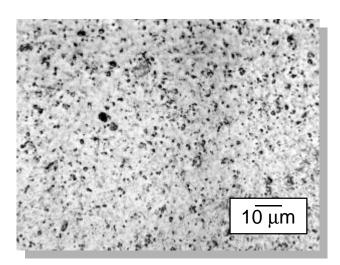
- Testing conducted at Ricerca using a Horiba LS-900 particle size analyzer
  - Unit capable of simultaneously measuring from 0.05 μm to 1020 μm diameter particles
- No powder was available for powder lot 3A

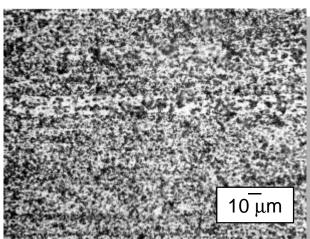
### **Extrusion**

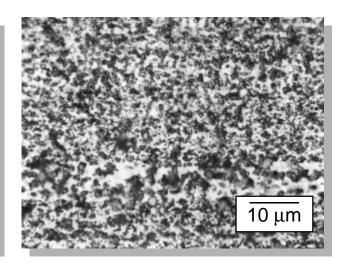


- Extrusions conducted at CSM Industries, Coldwater, MI
- X-ray pole figures showed minimal [111] texture and no [100] texture

# Optical Micrographs Of Extruded Cu-8 Cr-4 Nb





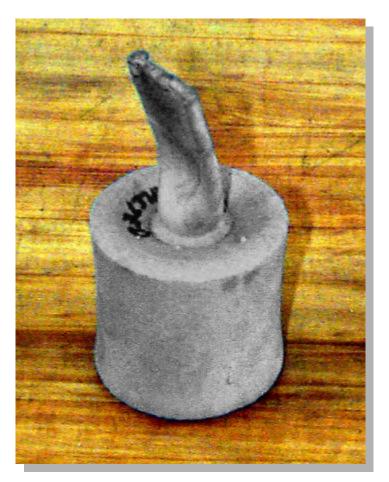


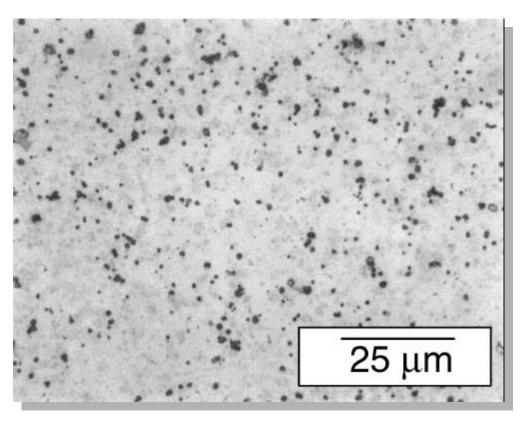
Transverse to extrusion direction

Longitudinal to extrusion direction

- Electropolishing reveals precipitates in relief extremely well
- Copper grain size may be too fine for optical microscopy
- Possible recrystallized grains observed in longitudinal crosssections where precipitate volume fraction is locally low
- Electropolishing techniques are still under development
  - Precipitates appear to be redepositing from electrolytic solution
  - Apparent volume fraction of Cr<sub>2</sub>Nb precipitates is higher than real volume fraction

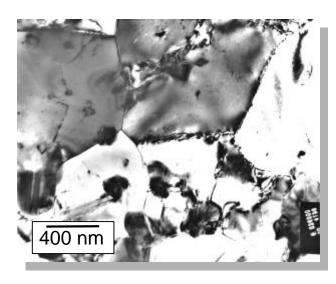
## **Hot Isostatic Pressing (HIPing)**



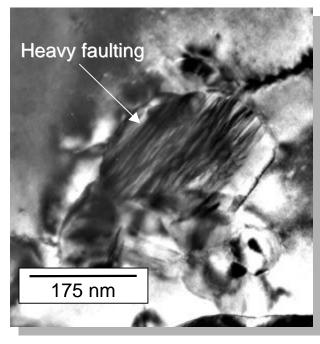


- Samples HIPed at 955°C (1750°F) to minimize Cr<sub>2</sub>Nb coarsening
- Full consolidation appears to have been achieved
  - Density measurements will be run on samples after machining

# Transmission Electron Micrographs Of Extruded Cu-8 Cr-4 Nb



175 nm



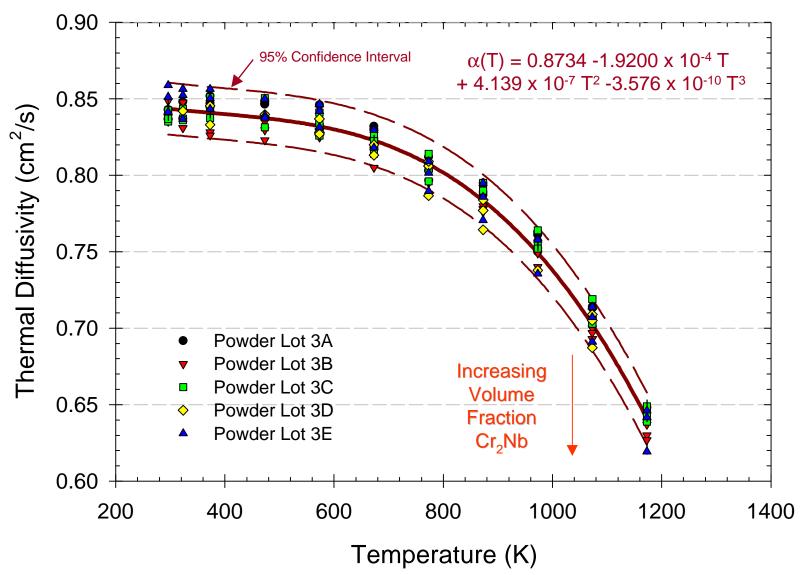
General Microstructure

Cr and Cr<sub>2</sub>Nb Precipitates

Faulting in Cr<sub>2</sub>Nb

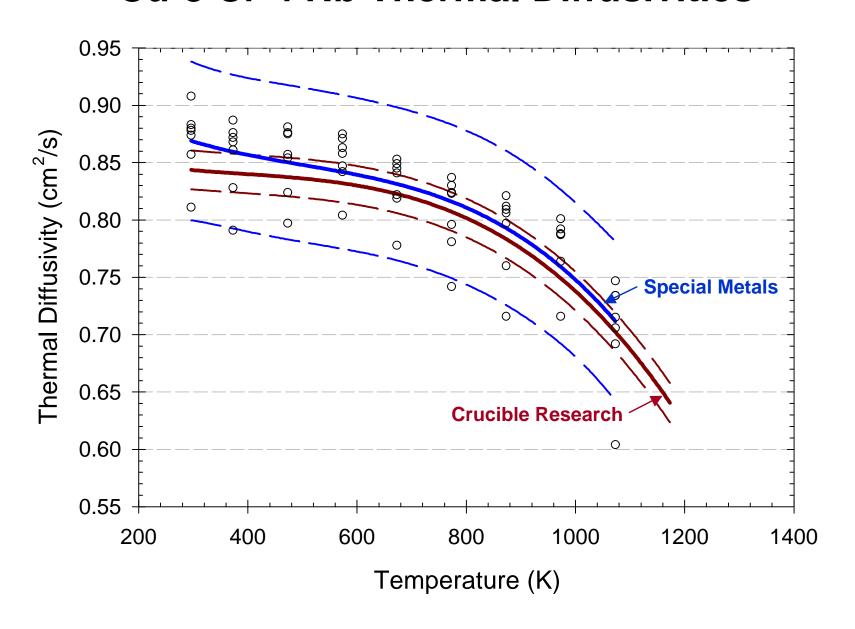
- Cr<sub>2</sub>Nb and Cr precipitates identified
- Cr<sub>2</sub>Nb precipitates have a very faulted structure
- Cr<sub>2</sub>Nb range from 150 to 850 nm
- HIP samples have nearly identical microstructures

### **Cu-8 Cr-4 Nb Thermal Diffusivity**

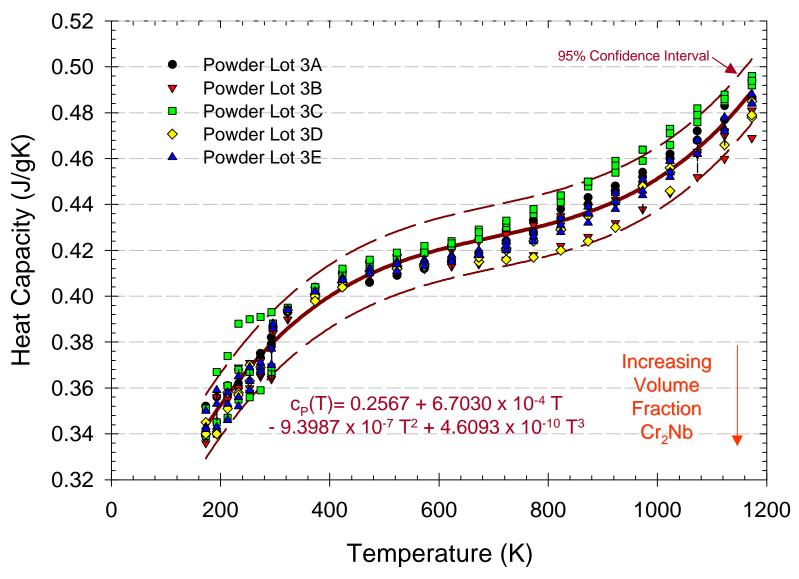


Statistical analysis did not show any discernable lot-to-lot variations in thermal diffusivity

# Comparison of Crucible and Special Metals Cu-8 Cr-4 Nb Thermal Diffusivities

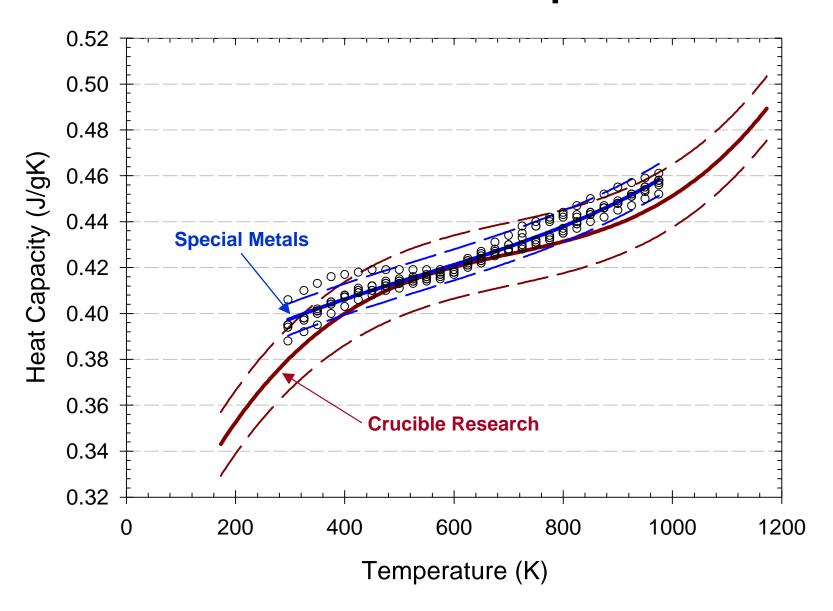


## Cu-8 Cr-4 Nb Heat Capacity

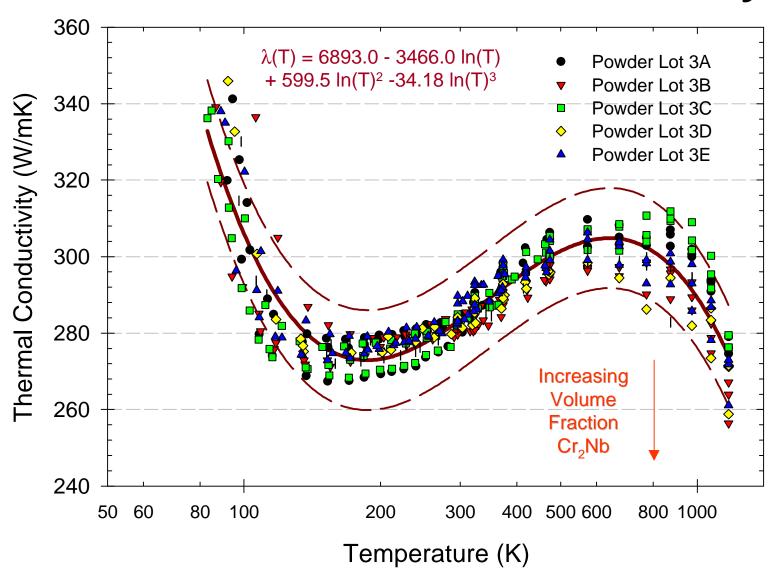


Statistical analysis of elevated temperature heat capacities indicates lot-to-lot variations in heat capacity

# Comparison of Crucible and Special Metals Cu-8 Cr-4 Nb Heat Capacities

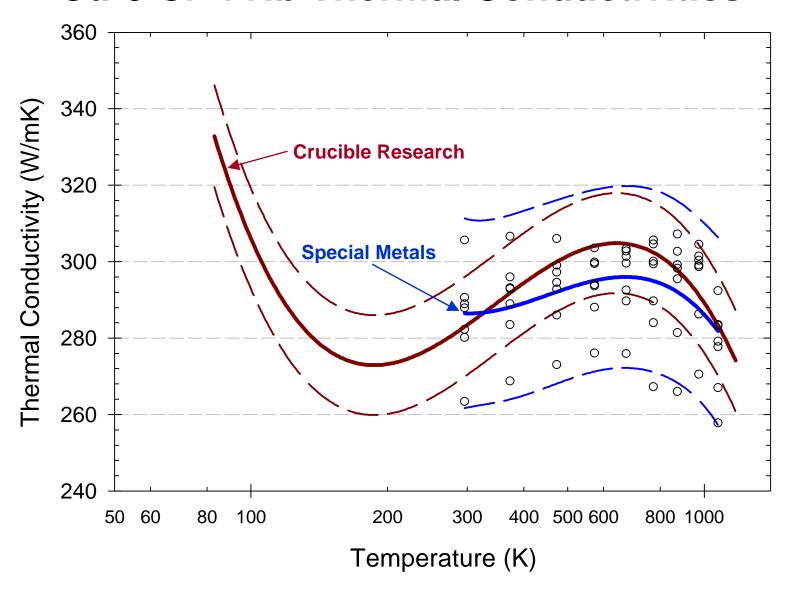


### **Cu-8 Cr-4 Nb Thermal Conductivity**

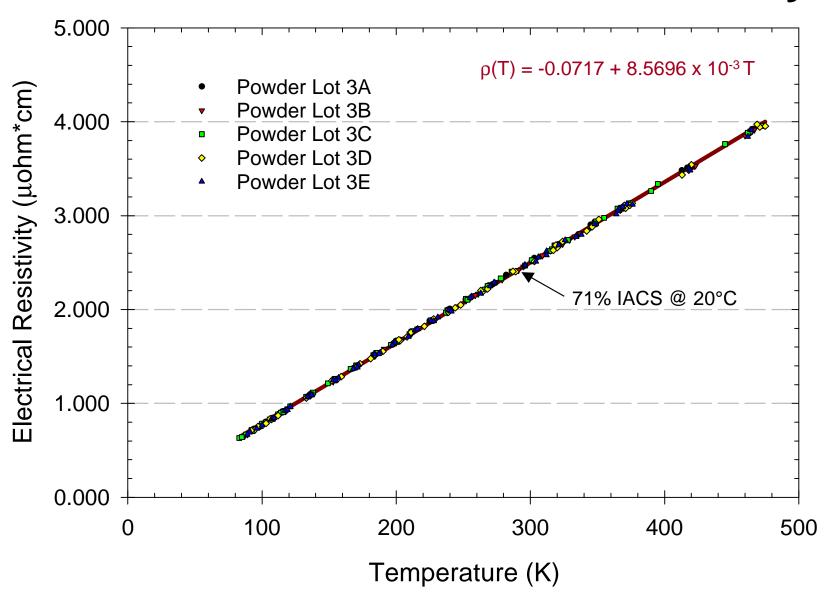


Statistical analysis of elevated temperature thermal conductivities indicates lot-to-lot variations in thermal conductivity

# Comparison of Crucible and Special Metals Cu-8 Cr-4 Nb Thermal Conductivities

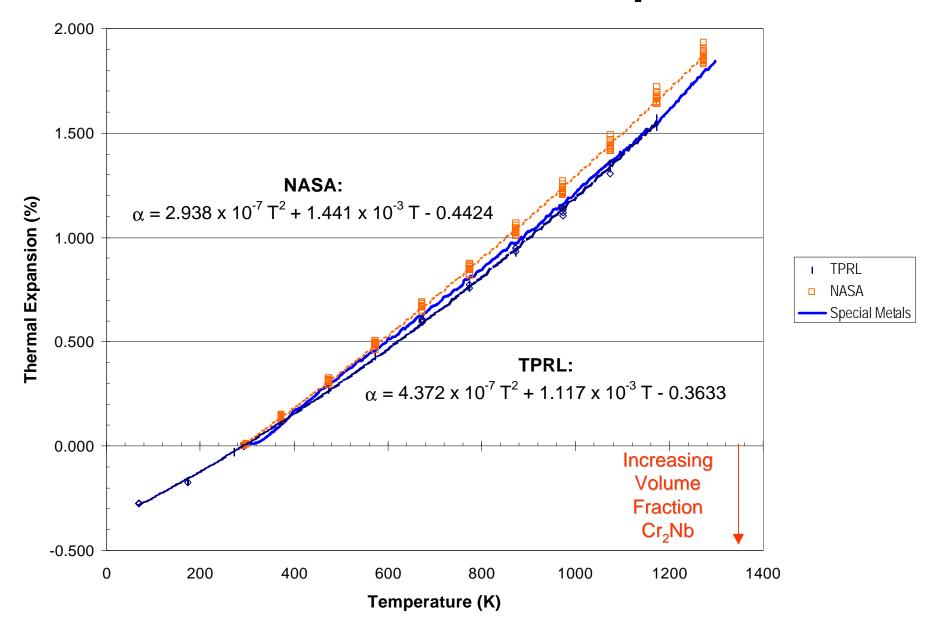


### **Cu-8 Cr-4 Nb Electrical Resistivity**



No discernable lot-to-lot variations in electrical resistivity

### **Cu-8 Cr-4 Nb Thermal Expansion**



### **Current and Future Work**

#### Tensile Testing

- Cryogenic tensile testing at MSFC completed
- Tensile testing between room temperature and 800°C currently being conducted

#### Creep Testing

- Short term (50 h max.) creep tests between 500°C and 800°C are currently being conducted
- Low Cycle Fatigue (LCF) Testing
  - Room temperature endurance limit of 0.5% strain range has been established in preliminary testing
  - Testing between room temperature and 400°C with 0.7% and 2% strain ranges is being started
- Fractography and Quantitative Microscopy
  - Fractography and related microscopy to be conducted as samples are tested
  - Quantitative microscopy to determine volume fractions of Cr<sub>2</sub>Nb, Cr<sub>2</sub>Nb size distributions and copper grain sizes will be done

### **Further Information**

Further information as it becomes available can be found at: polly.grc.nasa.gov/MDWeb/People/MSELLIS.html